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THE INCUBATOR TEST FOR SEWAGE EFFLUENTS.

By F. Wallis Stoddart, F.I.C.

The incubator test stands alone amongst the various means devised for determining the quality of a sewage effluent, in that it affords a direct and conclusive proof of conformity, or the reverse, with the requirements of the Rivers Pollution and Public Health Acts. Offences against these Acts, with the exception of the improper deposition of solids, are the result of putrefactive changes. The ordinary analysis of an effluent and its relation to dissolved oxygen afford valuable but only indirect evidence of its liability to putrefaction, the deductions drawn from the analytical data being further based upon premises not universally accepted, whilst the analysis itself is far too cumbrous a performance to apply sufficiently often to give a reliable insight into the character of so variable a liquid as sewage effluents commonly are; the incubator test, on the other hand, goes at once to the root of the matter, can be applied to any reasonable number of samples, and gives results which require no further elaboration.

The method of carrying out this test is thus described in the "Experts' Report on Treatment of Manchester Sewage": "A determination is first made of the oxygen absorbed from potassium permanganate by the sample in three minutes. A bottle is then completely filled with the sample, and placed in the incubator at 80° F. for six or seven days. The three minutes' oxygen absorption is then again determined. If any putrefaction has taken place, the oxygen absorbed in three minutes will exhibit a decided increase in amount owing to the more ready oxidizability of the products of putrefaction, such as sulphuretted hydrogen, etc. On the other hand, if the sample keeps sweet, the three minutes' oxygen absorption remains practically unchanged after incubation, or there will be a slight decrease owing to slight oxidation of the impurities which has taken place during the period of incubation at the expense of the nitrate or dissolved air present in the sample.

The test under these conditions is not entirely satisfactory, and does not distinguish with sufficient sharpness between good and bad effluents, as may be seen on reference to the Manchester report, in which the increase in oxygen absorption by unfiltered tank effluent rarely reaches 50 per cent., and even the crude sewage occasionally shows complete stability.

With the view of improving this test, and reducing, if possible, the length of

incubation, I have carried out a number of experiments with sewage products of various kinds, and, chiefly by raising the temperature of incubation, have succeeded in so far emphasizing the distinction between putrescible and imputrescible liquids as to have arrived at a test which might well be adopted in connection with any future legislative measures. The liquid to be tested should be enclosed in well-stoppered bottles, which should be quite full. Mercury valves are objectionable, as the mercury is attacked and fixes an appreciable amount of the sulphides formed in putrefaction. The incubating temperature should be 37° C., when much more active changes will ensue than at 27° C., resulting in a much more copious production of reducing bodies. The bottle should be well shaken before the measured quantity is taken for the estimation of absorbed oxygen.

With unfiltered tank effluent a marked increase in absorption is noted after three days, but the change is much more pronounced at the end of a week, and continues for a considerably longer period.

It is found that the formation of sulphide runs parallel with, and is no doubt the chief cause of, the increase in oxygen absorption; this being so, the test may be still further simplified by substituting the addition of lead solution for the permanganate process, and, if necessary, a colorimetric estimation may be made, for which purpose a solution of lead chloride will be found to be preferable to the usual acetate. For all practical purposes, however, a visible darkening on addition of the lead test after incubation for from three to six days is all that is required, and this is the form of criterion I should suggest for official purposes; any effluent that will give evidence of putrescibility under the current test will be readily detected in this way. Indeed, the modified test is, if anything, too severe, and will be found to condemn many effluents which comply with the usual standards; as, however, it is now possible by modern methods to continuously convert sewage into a liquid capable of passing the modified test at a single expenditure of a few shillings per head of population, no great injustice would be inflicted by its adoption.

The following table illustrates the application of this test to unfiltered precipitation and septic effluents:

INCUBATOR TEST.

Three Minutes' Oxygen Absorption of Unfiltered Precipitation and Septic Effluents before and after Incubation at 37° C. in full Bottles.

Length of Incubation in Days.	Fresh.	1.	2.	3.	4.	5.	7.	8.	9.	10.	11.	12.
Precipitation effluent ...	0·700	0·700	0·727	0·952	1·30	1·40	1·456	1·624	1·638	1·68	1·736	1·960
Percentage increase ...	—	—	12	36	86	100	108	132	134	140	148	180
Septic effluent	0·560	0·560	0·694	1·12	1·68	1·89	1·994	2·036	2·13	2·156	2·268	2·436
Percentage increase ...	—	—	24	100	200	236	256	264	280	285	305	335

The formation of sulphide as indicated by the lead test is exactly parallel with the oxygen absorption.

It would be anticipated on theoretical grounds that the septic product would be a more putrescible and offensive liquid than the precipitation effluent, and that if treatment were limited to tank operations, as is even now sometimes suggested, chemical purification would be more effective than septic action alone. It is interesting to note the more copious and rapid production of oxidizable (and offensive) bodies in the much weaker septic effluent in the above table, a change which, however, presents marked advantages where the tank product is applied to the aerating filter now universally recommended.

COMPOUND TINCTURE OF BENZOIN.

By Alfred Hill, M.D., F.I.C., and J. F. Liverseege, F.I.C., Ph.C.

The British Pharmacopœia of 1898 orders that compound tincture of benzoin shall be prepared from benzoin, prepared storax, balsam of tolu, Socotrine aloes, and 90 per cent. alcohol.

Benzoin.—The requirement of the Pharmacopœia is that benzoin shall be "almost entirely soluble in alcohol (90 per cent.)." The best quality of benzoin is Siam, and, according to Dieterich (*Chemist and Druggist*, November, 1898), from 95 to 100 per cent. is soluble in alcohol. Sumatra benzoin contains from 7·1 to 31·9 per cent. of insoluble matter (Evans, *Pharmaceutical Journal*, May, 1898; Dieterich, *Helfenberger Annalen*). The editor of the Pharmacopœia, in his 1898 Report, remarks:

"For official use practically no such insoluble matter is permissible; benzoin must be 'almost entirely soluble.' Until it has been shown that the bark, etc., has parted with harmful soluble matter to the alcohol used in preparing the official compound tincture, benzoin containing the usual varying proportions of bark (1 to 30 per cent.) may be employed, but allowance must, of course, be made for the insoluble matter, so that 1 pint of the official tincture shall be prepared from 2 ounces of *benzoin*, and not from 2 ounces of benzoin and bark."

Twelve samples of Sumatra benzoin examined by Dieterich lost from 4·6 to 8·2 per cent. by drying at 100° C. A single sample examined by us, after solution in alcohol, lost 4·6 per cent. of its weight on drying in the water-oven.

From the above results it seems reasonable to expect that in the preparation of the tincture 100 parts of official benzoin should yield soluble matter equivalent to at least 85 parts of dry extract, allowing a loss of 15 per cent. of insoluble and volatile matter.

Storax.—Crude storax contains from 1·4 to 7·3 per cent. of matter insoluble in alcohol, and from 19·6 to 41 per cent. of water (Dieterich, *Pharm. Central.*, 40, 427). The Pharmacopœia preparation is obtained from this "by solution in ethylic alcohol, filtration, and evaporation of the solvent." It is officially required to give off no

moisture on heating in a test-tube. Eight samples of prepared storax examined by Dieterich lost from 5·1 to 12·6 per cent. by drying at 100° C. He considers that the moisture should not exceed 8 per cent. (*Chemist and Druggist*, July, 1898). The United States Pharmacopœia requires that storax, after solution in alcohol and evaporation of the solvent, shall yield at least 70 per cent. of solid residue. As the British Pharmacopœia article has been purified, probably 80 per cent. of it will appear as dry extract when used for the tincture.

Balsam of Tolu.—The British Pharmacopœia requires that it shall be soluble in alcohol. Five samples examined by Dieterich conformed with this requirement. A sample examined by us after solution in alcohol lost 6·2 per cent. of its weight on drying at 100° C. Probably an allowance of 10 per cent. for volatile matter would be sufficient.

Socotrine Aloes.—The Pharmacopœia requires this drug to be almost entirely soluble in 45 per cent. alcohol. Hager (ANALYST, x. 95) states that it is completely soluble in 80 per cent. alcohol, but only partially in 90 per cent. When a gramme of Socotrine aloes was treated with 50 c.c. of 90 per cent. alcohol, as in making the tincture, and filtered, we found that the filtrate, after evaporation of the solvent and drying in the water-oven for three hours, yielded 97 per cent. of the weight taken in the form of dry extract. Probably, therefore, an allowance of 10 per cent. for insoluble and volatile matter is ample.

Quantity of Extract in the Tincture.—In the following table the quantities of the ingredients ordered by the Pharmacopœia for 1 litre of the tincture are given, and by the use of the standards given above the amount of the solid extract which should be yielded by each is calculated:

Quantities given by the Pharmacopœia.		Amount of Extract in	
		Drug.	Tincture.
	Grammes.	Per Cent.	Grammes.
Benzoin ...	100	85	85
Prepared storax ...	75	80	60
Balsam of tolu ...	25	90	22·5
Socotrine aloes ...	18·3	90	16·5
Per litre ...	218·3		184

We think that the above statement justifies the adoption of a standard of 180 grammes per litre as a reasonable one. It must be remembered that deficiency in one drug may be compensated by the good quality of the others. A tincture prepared by us without any special attention being given to the quality of the drugs contained 182 grammes of solid extract per litre. The extract was determined by evaporating 5 c.c. of the tincture to dryness on the water-bath in a German-silver dish 3 inches in diameter, with a flat bottom, and drying the residue three hours in the water-oven.

Twenty-five samples of compound tincture of benzoin purchased officially by the

Birmingham Food and Drugs Inspector during the last two years yielded the following amounts of solid extract:

Grammes Solid Extract Per Litre.	Number of Samples.	
201	1	
180 to 187	13	
174 to 178	7	
169	1	
158	1	
147	1	(Vendor was fined £5)
115	1	(Vendor was fined £20)

The average of the whole of the samples is 176 grammes of solid extract per litre, or, if the two obviously adulterated ones be omitted, 180 grammes. The sample with 158 grammes was passed as of low quality. Probably 160 grammes may be taken as the minimum limit for tincture of benzoin.

The standard of 180 grammes per litre is advocated by Moor and Priest (*Chemist and Druggist*, July, 1900). A sample prepared by Seyler (Report to the Glamorgan County Council, 1897) yielded 175 grammes. Merson, in a paper read this year before the Pharmaceutical Society, states that as a result of eight experiments, using all grades both of benzoin and storax, he considered not less than 170 to 180 grammes of solids per litre should be present. His mean result was 178 grammes. He points out that if storax is used, instead of the official *prepared storax*, less than 160 grammes may be present even if good Siam benzoin is used, and that a tincture prepared with a moderate Sumatra benzoin and prepared storax gave 181·5 grammes of solid matter per litre (*Pharmaceutical Journal*, February 23). At the recent meeting of the British Pharmaceutical Conference, H. Whippell Gadd stated that if the extractive falls much below 180 grammes per litre, it is evident that an inferior benzoin or storax, or both, have been used. F. W. Fletcher (*Chemist and Druggist*, January, 1901) gives 15·8 grammes as the "percentage of solid residue." If by "percentage" he means grammes per 100 c.c. the result is very low for a standard tincture, but if it is a true percentage, as the specific gravity of the tincture is given as 0·892, it will indicate 177 grammes per litre.

Specific Gravity of the Tincture.—The specific gravity of the tincture prepared by us was 0·896 at 60° C. The specific gravities of the twenty-five official samples were as follows:

Specific Gravity at 60° F.	Number of Samples.	
0·906-0·904	3	
0·903-0·900	8	
0·899-0·895	9	
0·893	2	
0·891	1	
0·886	1	(Vendor was fined £5)
0·880	1	(Vendor was fined £20)

In the next table are given various published specific gravities of the 1898 compound tincture of benzoin.

Specific Gravity.	Reference.
0·893	J. C. Umney. *Chemist and Druggist*, April, 1898.
0·894	J. Attfield. Report on Pharmacopœia for 1898.
0·900	J. Barclay. *Chemist and Druggist*, December, 1898.
0·902-0·907	("Allowance made for insoluble matter in the benzoin to accord with the British Pharmacopœia monograph, otherwise tincture is lighter.") Holthouse and Harvey. *Chemist and Druggist*, July, 1900.
0·890-0·900	Moor and Priest. *Chemist and Druggist*, July, 1901.
0·890-0·900	Year-Book of Pharmacy, 1900.
0·892	F. W. Fletcher. *Chemist and Druggist*, January, 1901.
0·8983	Merson. *Pharmaceutical Journal*, February, 1901.

The foregoing results are of considerable importance, in view of a case which has come before the magistrates at Birmingham. A sample of Pharmacopœial tincture of benzoin purchased under the Sale of Food and Drugs Act showed in two determinations a specific gravity of 0·8840 and 0·8846, and 146 and 147 grammes per litre. A certificate was given by the Public Analyst to the effect that the sample contained only 82 per cent. of the quantity of solid matter ordinarily present in tincture of benzoin. The inspector's sample having been referred to the chemists of the Government Laboratory, a report was received from them stating that "in our opinion it affords no evidence of being below the strength of the compound tincture of benzoin made according to the British Pharmacopœia of 1898." By the courtesy of the Chief of the Government Laboratory, we are enabled to state that the sample in question was found by the Government chemists to contain 133 per cent. of proof-spirit by volume, and yielded 16·4 per cent. of extract dried at 100° C. Calculation shows that 147 grammes per litre is equal to 16·6 per cent. by weight, which is in practical agreement with the quantity found at the Government Laboratory. Reference to the figures given in the earlier portion of the paper shows that the quantity should be considerably higher than this.

THE RECIPROCAL DETERMINATION OF CYANIDES AND CYANATES.

By J. W. Mellor, B.Sc. N.Z. University.

Enormous quantities of potassium cyanide are used in certain gold-extraction processes. It is well known that the commercial article contains more or less cyanate, which appears to be useless from a metallurgical point of view. In the course of some experiments on solutions of potassium cyanide, it was desired to estimate the quantity of potassium cyanate in the presence of cyanide and *vice versâ*.

F. Wöhler* has pointed out that silver cyanate is decomposed by dilute nitric acid thus:

$$AgOCN + 2HNO_3 + H_2O = AgNO_3 + NH_4NO_3 + CO_2.$$

The addition of nitric acid before the usual titration, however, interferes with the accuracy of the test, since the double cyanide of silver and potassium is decomposed

* Wöhler: Gilbert's *Ann. d. Phys.*, 73, 157, 1823.

by the dilute acid, with evolution of hydrogen cyanide and the precipitation of silver cyanide.

A. H. Allen* has employed Wöhler's reaction for the determination of cyanates Since 1 molecule of silver cyanate neutralizes 2 molecules of nitric acid, if excess of normal acid be added to the cyanate, on titrating back with normal alkali, 1 c.c. normal HNO_3 neutralized by the cyanate represents 0·0405 gramme potassium cyanate.

G. Denigés† has improved the ordinary cyanide determination by Liebig's process, by titrating an ammoniacal cyanide solution with $\frac{N}{10}$ $AgNO_3$, using 10 drops of a 20 per cent. aqueous solution of KI as indicator. The sparing solubility of AgI in aqueous ammonia makes the end-reaction much sharper. As usual, one equivalent of $AgNO_3$ represents two equivalents of KCN.

Since silver cyanate and cyanide are both much more soluble in aqueous ammonia than silver iodide, Denigés' process may be applied. The presence of KOCN appears to make no difference to the cyanide determination under these conditions—*e.g.* :

> Ten c.c. KCN solution required 14·3 c.c. $\frac{N}{10}$ $AgNO_3$; 10 c.c. of the same solution, with 10 c.c. of KOCN solution added, required 14·3 c.c. $\frac{N}{10}$ $AgNO_3$; and similarly with 40 c.c. of the same solution of KOCN added, 14·4 c.c. $\frac{N}{10}$ $AgNO_3$ was necessary.

Thus, Denigés' and Allen's processes are reciprocal for the determination of cyanates and cyanides in the presence of one another.

The principle of the proposed method for the determination of cyanides and of cyanates is first to determine the equivalent of the cyanide in terms of silver nitrate, and then to estimate the amount of cyanate in the whole in terms of nitric acid.

The following *modus operandi* was adopted with good results :

1. Dissolve 20 grammes commercial potassium cyanide in about 100 c.c. of distilled water. Add a solution of calcium nitrate—free from chlorides—to precipitate any carbonates that may be present. Barium nitrate cannot be used on account of the insolubility of barium cyanate. Filter and wash the precipitate with distilled water. Collect the filtrate and washings in a 200 c.c. flask, and make up to 200 c.c. One c.c. of this solution represents 0·1 gramme of the original sample.

The precipitate contains insoluble impurities (carbonaceous and earthy matters), carbonate and sulphate of calcium, etc. If desired, each of these may be determined by the usual methods of analysis.

2. Mix 10 c.c. of the above solution of the sample of cyanide with excess of aqueous ammonia and a few drops of KI solution. Titrate with $\frac{N}{10}$ $AgNO_3$ solution, and suppose that *m* c.c. of this solution are required.

3. Run concentrated $AgNO_3$ solution into another portion—say, 10 c.c. of the above aqueous solution of cyanide—until no further precipitation occurs. Thus, all the cyanide radicle in the $KAgCy_2$ is precipitated as AgCN. Filter and wash the precipitate of mixed cyanide and cyanate of silver with ice-cold distilled water. Add,

* Allen's "Commercial Organic Analysis," 1896, 3, iii., 484. This observation appears to be the only published record on the determination of cyanates in the presence of cyanides.

† Denigés, *Ann. de Chim. et de Phys.* [7], 6, 381, 1895. An abstract occurs in the *Journ. Chem. Soc.*, 1896.

say, 5 c.c. normal HNO_3 and warm to about 50°, when any cyanate will be decomposed. Filter and wash. Treat the filtrate with standard alkali. Suppose that n c.c. of normal NaOH solution are required, then 0·0405 $(5-n)$ gramme (or 4·05 $[5-n]$ per cent.) KOCN are contained in 1 gramme of sample; 0·013 m gramme (or 1·3 m gramme per cent.) KCN are contained in 1 gramme of sample.

If desired, the results can be expressed as "available cyanogen"; from 0·0052 m gramme (or 0·52 m gramme per cent.) available cyanogen are contained in 1 gramme of sample.

Instead of using 10 c.c. of the original solution in 3 above, q c.c. may be used when there is 0·00405 $(5-n)$ q gramme KOCN in 1 gramme of sample.

If n' c.c. of $\frac{N}{10}$ alkali be used, then 10 n' must be substituted for n unless $\frac{N}{10}$ acid has also been employed.

If p c.c. of nitric acid be used instead of 5 c.c. in the decomposition of silver cyanate, then there is 0·0405 $(p-n)$ gramme KOCN in 1 gramme of sample.

The presence of sodium cyanide introduces a considerable error in the calculation of available cyanogen to potassium cyanide alone. The maximum amount of available cyanogen in pure KCN is 40 per cent., while for NaCN the maximum is 53 per cent.; hence a little of the latter present would calculate to more KCN than was actually present. A sample containing 15 per cent. NaCN would be reported as 100 per cent. KCN.* This would be an unsatisfactory valuation unless sodium cyanide is at least equal to the potassium salt in its power of extracting gold from "tailings."

Chlorides, if present, may be estimated as follows: Fuse in a porcelain basin a known weight of sample, with a mixture of KNO_3 and Na_2CO_3 (1 : 5) carefully added. Boil the fused mass with water acidulated with HNO_3, and estimate the chlorides in solution in the usual way.†

The following is an analysis made during October, 1897, in the Otago University College Laboratory, New Zealand, of a specimen obtained from a gold extraction company such as was in use in their cyanide works. No sulphides, thiocyanates, or formates were present:‡

	Percentage Amounts.
Insoluble matter { Carbonaceous	4·35
Insoluble matter { Ash	1·07
Carbonate radicle (CO_3)	4·69
Sulphate radicle (SO_4)	traces
Chloride radicle (Cl)	traces
Cyanate radicle (OCN)	4·04
Cyanide radicle (CN)	32·51
Potassium	49·90
Sodium	1·59
Loss	1·85

The loss is probably due to moisture, although the specimen stood over $CaCl_2$ for twenty-four hours before weighing. It seems impossible to determine to which

* Kayser, *Chem. Zeitung*, 16, 1148, 1892. † Watts' "Dictionary of Chemistry," 1889, 346.
‡ *Ibid.*; *cf.* also Denigés, *loc. cit.*

negative radicles Na or K must be calculated; hence the advantage of recording results as "available cyanogen."

With artificially-prepared mixtures of "pure" potassium cyanide by Wiggers' process,* and of potassium cyanate by Bell's process,† an average error of $+0{\cdot}18 \pm 0{\cdot}01$ per cent. in the determination of KOCN was obtained when the components of the mixture were assumed to be absolutely pure. The constancy of the error, however, would make this appear doubtful. More recent determinations with KOCN and KCN of known purity gave an average error of $0{\cdot}04 \pm 0{\cdot}01$ per cent. in ten experiments.

SYSTEMATIC INSPECTION OF MILK FOR PRESERVATIVES.

By Albert E. Leach, S.B.

(*Analyst of the Massachusetts State Board of Health*).

In the June number of the Analyst for the current year (p. 148) appeared a paper by M. Wynter Blyth on "The Detection and Estimation of Preservatives in Milk," in which was described a method for subjecting all the samples to certain fixed bacterial conditions during a period of some twenty-four hours, with a view to selecting those samples which contained preservatives, and afterwards identifying the particular preservative present.

It occurred to the writer that it might not be without interest in this connection to English analysts to know how, as a matter of routine, a large number of milk samples are systematically subjected to tests for preservatives daily throughout the summer in the laboratory of the Department of Food and Drug Inspection of the Massachusetts State Board of Health.

All the milk samples collected by three inspectors throughout the State, with the exception of four western counties, are analysed in the laboratory of the Department at Boston. These amount to upwards of 500 samples per month, the daily number analysed varying from 10 to 60, and during the months of June, July, August, and September, in addition to the regular analysis for total solids and fat, all are systematically examined for preservatives by methods which during four years have proved both quick and accurate.

At the outset it should be said that the only preservatives found in milk in Massachusetts at the present time are formaldehyde, boracic acid and borax, and sodium bicarbonate. Salicylic acid has not been found in milk in this State for many years. It is a very poor milk preservative, on account of the taste imparted to the milk, and it is difficult to conceive why it should be used at all at the present time for this purpose, in view of the much more efficient antiseptics at hand.

The following summary shows the extent of the use of antiseptic substances in milk in Massachusetts during the last three years:

* Wiggers, *Liebig Ann.*, 1839, 29, 65. † Bell, *Chem. News*, 1875, 32, 99.

Year.	Samples Examined.	No. containing Formaldehyde.	Per Cent. containing Formaldehyde.	No. containing Boracic Acid.	Per Cent. containing Boracic Acid.	No. containing Carbonate.	Per Cent. containing Carbonate.	Total containing Preservative.
1898* ...	1,046	26	2·5	11	1·0	4	0·4	41
1899† ...	2,105	55	2·6	13	0·6	3	0·3	71
1900† ...	2,018	61	3·0	6	0·3	—	—	67
Total	5,169	142	2·7	30	0·6	7	0·1	179

Thus, out of 5,169 samples examined for preservatives, 179, or 3·5 per cent., were found to contain them. It will be seen from the above table that the use of boracic acid and sodium bicarbonate is on the decrease, while formaldehyde is being used more and more. This is to be expected, in view of the fact that formaldehyde, being in solution, lends itself more naturally to the milkman's immediate use, besides being the most active of preservatives, and imparting no appreciable taste to the milk, even when used in considerable strength.

The method of procedure is as follows: After the milk samples have been analysed for total solids and fat, they are arranged in order, and about 10 c.c. of each sample is poured into a porcelain basin, the basins being, for convenience, arranged in the same order as the containers of the milk samples. Each casserole is then treated alike in succession, first adding, from a large stock-bottle provided with a delivery siphon and stopcock, about 10 c.c. of the hydrochloric reagent, and then heating directly over the gas-flame, holding the basin by the handle, and giving it a rotary motion during the heating to break up the curd and hasten its solution in the acid. The reagent contained in the stock-bottle consists of commercial hydrochloric acid (specific gravity, 1·20) containing 1 c.c. of 10 per cent. ferric chloride solution to each 500 c.c. of acid. The heating is continued nearly to the boiling-point. The presence of formaldehyde is indicated by the well-known violet coloration, varying in depth with the amount present. By this test it has been found that 1 part of formaldehyde in 500,000 parts of milk is readily detected before the milk has soured. After souring, the limit of delicacy proves to be about 1 part in 50,000. In point of actual time consumed in making the test, less than one minute is sufficient for each sample.

The hydrochloric acid method has been the subject of much criticism, it being alleged that substances in milk other than formaldehyde produce colour reactions. Many analysts prefer to use sulphuric acid for the test, but the latter is far less delicate, because of the charring that inevitably results by the action of the acid on the milk. The reliability of the hydrochloric acid method has been tested again and again in this laboratory, and, while it may not be considered best to rely on this test as the sole evidence of formaldehyde for purposes of a court case, but rather to employ it as a preliminary test, it can nevertheless be said that the writer has never known of a single instance in which the presence of formaldehyde as indicated by the colour reaction with hydrochloric acid was not confirmed by subsequent tests, such,

* Samples examined for preservatives during July and August only.
† Samples examined for preservatives during June, July, August, and September.

for instance, as testing the first few drops of the distillate from the milk with Schiff's reagent, or with the mixture of resorcin and sulphuric acid recommended by Mulliken. Various aldehydes, when present in milk, give colour reactions when treated with hydrochloric acid as above, but nothing but formaldehyde gives the peculiar violet coloration which is perfectly distinguishable and unmistakable to the practised eye.

The preliminary tests for boracic acid and carbonate require even less time than that for formaldehyde, being made in each case in the original platinum dish in which the 5-gramme portion for total solids was weighed out. In fact, the tests are so arranged as to be, as it were, only a side-issue in the process of cleaning the platinum dishes. The dishes are, of course, numbered, and the residues from the total solids are incinerated to an ash in each dish one after another. The dishes containing the milk ashes are then arranged side by side, and, by means of a pipette, 1 or 2 drops of dilute hydrochloric acid are introduced into each in succession, taking notice whether effervescence ensues, which in itself would indicate the presence of carbonate, and is very perceptible when the carbonate is present in as small amount as 0·05 per cent. In case of effervescence, the corresponding sample of the original milk is further examined by the rosolic acid test for carbonate.

Continuing, the test for boracic acid is carried out by adding with a wash-bottle a few cubic centimetres of water to the acidulated ash of each milk sample in the dishes, moving each dish slightly to get the ash into solution, and finally placing in each solution a strip of turmeric paper. Formerly it was customary to at once place these strips of turmeric paper on parallel glass rods for drying, the arrangement being such that each strip of paper occupied a numbered position corresponding to the number of the platinum dish from which it was taken; but of late it has been found simpler and just as efficient, having dipped each strip in the solution, to allow it to adhere to the side of the original dish, and remain there until dry. If the paper, when dry, is of a deep cherry-red colour, turning a dark olive when treated with a dilute solution of sodium hydroxide, the presence of boracic acid is assured.

These rapid methods, if carefully conducted to avoid confusion of samples, are serviceable in indicating at least, in a preliminary way, the samples containing added preservative, as well as the preservative employed, and in all cases as many confirmatory tests as desired may be conducted.

THE DETECTION OF STEMS IN GROUND CLOVES.

By Edgar B. Kenrick.

It would appear that the green colouring matter (chlorophyll) is absent from the clove-buds, while the stems contain it in considerable quantity. To detect stems—a common adulterant in ground cloves—the following simple method may be used: Shake up a small quantity of the material in a test-tube with alcohol, allow the sediment to settle, and examine the absorption spectrum of the tincture for the characteristic bands of chlorophyll. Nearly all samples of whole cloves contain, when purchased, a certain proportion of stems. These may be picked out, ground up, and used for purposes of comparison.

University of Manitoba, Winnipeg, *October* 3, 1901.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

Recognition of Boiled Milk. Kühnau. (*Milch Zeit.*, 1901, xxx., 327; through *Chem. Zeit. Rep.*, 1901, 184.)—The author recommends Arnold's test with guaiacum tincture for this purpose; but it is important that the light-brown tincture of guaiacum wood be employed, not the resin tincture or an ammoniacal product. The tincture of the wood should be tested before use: 1 part mixed with 15 of raw milk should quickly yield a dark-blue colour which soon disappears, and comes again on adding more reagent. Boiled milk, or a sample heated above 80° C., only gives a dirty yellow colour. F. H. L.

The Detection of Cocoanut Oil in Cocoa Butter and Chocolate. J. Wauters. (*Bull. de l'Assoc. Belge*, 1901, xv., 131, 132.)—This is readily effected by the author's distillation method (ANALYST, xxvi., 128), as is shown by the following results obtained with pure samples of the respective fats:

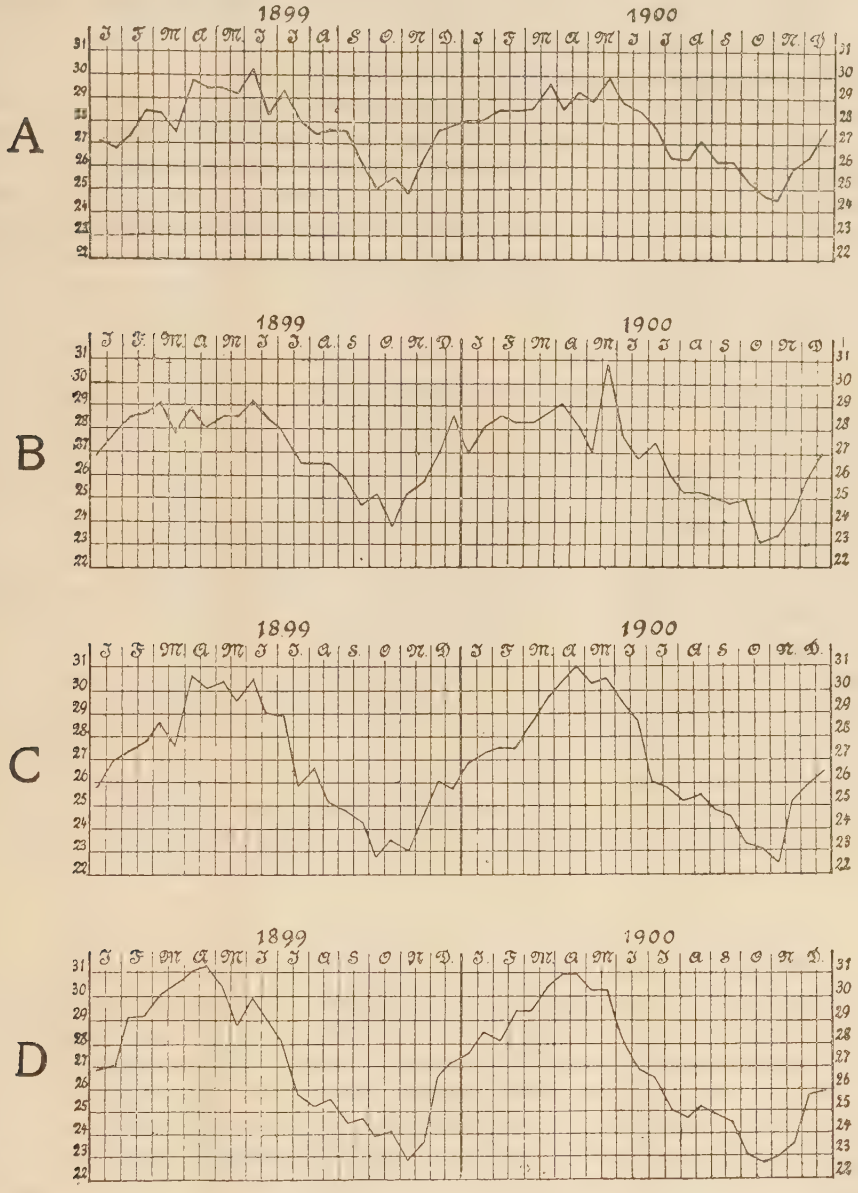
	Soluble Volatile Acids.			Insoluble Volatile Acids.		
	1st Distillation.	2nd Distillation.	Total.	1st Distillation.	2nd Distillation.	Total.
Cocoa butter ...	0·10	0	0·10	0·25	0·15	0·4
Cocoanut oil ...	7·10	4·30	11·40	7·85	7·55	15·4

C. A. M.

The Halphen Reaction and American Lard. P. Soltsien. (*Zeits. öffentl. Chem.*, 1901, vii., 140; through *Chem. Zeit. Rep.*, 1901, 167.)—The author has prepared samples of fat from American pigs which had been fed on cottonseed cake. The material was soft and of a brownish-yellow colour. With the Halphen test it gave a colour suggesting the presence of more than 0·25 per cent. of cotton oil. The nitric acid test gave even a stronger indication. Becchi's test also succeeded, though more faintly, but the Welman test for cotton oil failed. The iodine value of the fat was rather high, 68, and therefore inconclusive. Apparently the only proof that cotton oil had not been added to the lard was the fact that phytosterin was absent. Soltsien therefore considers that if a lard fails to give the Halphen reaction, less than 0·25 per cent. of cotton oil is present, and the sample need not be examined further; but if the Halphen test succeed, phytosterin must be sought for. Probably the active material in the Halphen and the nitric acid tests is identical. F. H. L.

The Reichert-Meissl Number for Butter. M. Siegfeld. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, 433; and **P. Vieth** (*Ber. Milchwirtschaft. Inst. Hameln.*, 1900.)—The authors have carried out a considerable number of investiga-

tions for the purpose of ascertaining the variations which the composition of butter undergoes during different times of the year. The butters from four North Hanover dairies were analysed twice a month during the whole of 1899 and 1900, and the



results given in the table show that the Reichert-Meissl number was highest (28 to 31·3) in the months of April, May and June, and lowest (22·4 to 26·4) in October and November of each year. The butters were from the mixed milk of about 5,000 cows.

The dairies from which they came were situated at Bülkau, Wesermarsch, Esens, and Georgsheil, and are designated A, B, C, D respectively in the tables. It will be noticed that the numbers obtained vary from 24·6 to 29·9 for A, 23·2 to 30·9 for B, 22·4 to 31·0 for C, and from 22·8 to 30·9 for D. In considering whether a butter is adulterated, it is suggested that the above facts be taken into consideration, a lower limit being taken during the months of October and November.

A. J. Swaving (*ibid.*, 1901, 577) comes to the conclusion that, in the late autumn, and principally due to poor feeding, the Reichert-Meissl number decreases whilst the Crismer number (the temperature at which the butter dissolves in absolute alcohol, under the conditions described by Crismer) rises. In the spring the reverse takes place. Variations in the food of the cows are quickly detected in the composition of the butter-fat. It is finally stated that the Reichert-Meissl number is not specially lowered in the autumn if the food of the cows be supplemented by feeding-cakes, etc. W. P. S.

A New Indicator for Use in Determining Total Acidity in Wines. E. G. Runyan. (*Journ. Amer. Chem. Soc.*, xxiii., 402.)—The author has applied to the estimation of the total acidity of wines, vinegars, and ciders an indicator proposed by L. Lachaux for estimating the alkalinity of beet-juices and molasses. The indicator is prepared by dissolving 3·1 grammes rosolic acid in 150 c.c. of 90 per cent. alcohol, neutralizing, and mixing with a solution of 0·5 gramme malachite-green in 50 c.c. of alcohol. With this mixture alkalies give a purple colour, changed to green by acids. The malachite-green is not affected, but renders the end reaction of the rosolic acid more distinct.

The titrations were carried out by diluting 10 c.c. of the sample with 300 c.c. of boiling water, heating for one minute to boiling to expel carbon dioxide, and cooling to 75°; after adding 10 drops of the indicator, an excess of decinormal alkali was added, and the excess determined with a standard acid solution. The results obtained agreed well amongst themselves, but were lower than those obtained by direct titration with standard alkali, using litmus and phenolphthalein as indicators, and the end-point was more easily determined than with these indicators.

A. G. L.

Estimation of Camphor in Camphor Oil. H. Löhr. (*Chem. Zeit.*, 1901, xxv., 292.)—Japanese oil of camphor contains two commercially valuable constituents—camphor and safrol. The latter is found in the dark oil, which has a specific gravity of a little more or less than 1·0. Since the density of this oil increases with its safrol content, it is valued by its specific gravity. The other variety of oil is valuable according to the amount of camphor it contains, but apparently no rapid and accurate process for the estimation of that substance has yet been published. The camphor is obtained from the oil by exposing it to a low temperature, the solid which separates being removed. Then the oil is distilled, and the fractions containing the camphor are treated similarly until the whole of that material has been recovered. The author's analytical process is an imitation of this method on the small scale:

At least 300 grammes of the oil are separated by distillation into three fractions: (*a*) Up to 195° C.; (*b*) between 195° and 220°; and (*c*) above 220° C. The middle fraction is placed for an hour in a freezing mixture, and the camphor which crystallizes out is collected by means of the pump. It is packed in filter-cloth, wrapped up in paper, and pressed for half an hour, then clean paper is applied, and the pressure repeated for fifteen minutes. The oily liquor is again distilled similarly four times, cooling the fraction passing over between 205° and 220°, and pressing only once for twenty minutes. The camphor recovered is finally weighed. If the original sample is very viscid, and therefore contains a large quantity of camphor, it should first have part of that constituent removed by simple freezing. Various examples are quoted which appear reasonably accurate.

Löhr mentions that two specimens of camphor oil were recently examined in a municipal laboratory in Japan by distilling them and collecting the fractions 195° to 215° C., which solidified on cooling. These were returned as "camphor chiefly," and the samples were stated to contain 54·13 and 64·58 per cent. of camphor respectively, whereas by Löhr's process the same oils gave 19 and 21 per cent. only.

F. H. L.

Oils of Sandalwood, Lavender, and Thyme. L. F. Kebler. (*Amer. Journ. Pharm.*, 1901, lxxiii., 223-227.)—*Sandalwood Oil.*—The physical and chemical constants generally accepted for a pure oil, which must readily dissolve in 70 per cent. alcohol, are: Specific gravity at 15° C., 0·970 to 0·978; optical rotation at 25° C. in a 100-millimetre tube, −17° to −19°; santalol, at least 90 per cent. Sample No. 1 in the subjoined table was prepared by the author from a wood yielding 5·5 per cent. of oil. Its constants fall well within these limits. The other samples were representative of the commercial oil. The author regards No. 6 as undoubtedly adulterated, and Nos. 3, 4, and 9 as inferior.

No.	Specific Gravity.		Santalol, Per Cent.	Optical Rotation.	Solubility in 70 Per Cent. Alcohol.	Santalol Esters, Per Cent.
	15° C.	25° C.				
1	0·9767	0·9724	97·16	−17°15′	1 in 5	3·06
2	0·9727	0·9707	93·64	−18°16′	,,	4·10
3	0·9747	0·9739	91·70	−14°56′	,,	2·93
4	0·9666	0·9601	90·12	—	,,	1·48
5	0·9716	0·9685	92·87	−17°2′	,,	1·43
6	0·9626	0·9600	75·00	− 7°4′	,,	2·67
7	0·9721	0·9681	96·34	−16°36′	,,	—
8	0·9713	0·9678	94·53	−16°56′	,,	3·61
9	0·9734	0·9696	90·87	−13°48′	1 in 5½	—

Oil of Lavender.—According to Gildemeister and Hoffmann, oils containing less than 30 per cent. of esters are usually adulterated. In the author's opinion, however, this statement is too sweeping, for genuine oils of lavender are known containing as little as 10 per cent. of esters, and yet highly esteemed commercially.

The four examples examined by the author were all considered to be of good quality. They gave the following results:

No.	Specific Gravity at 15° C.	Solubility in 70 Per Cent. Alcohol.	Optical Rotation.	Esters, Per Cent.
1	0·8985	1 in 3	−6°6′	25·70
2	0·8989	,,	−2°54′	34·36
3	0·8892	,,	−5°9′	31·42
4	0·8830	,,	−3°41′	28·29

Oil of Thyme.—The author gives the following as the characteristics of the genuine oil: Soluble in 1 to 2 vols. of 80 per cent. alcohol; specific gravity at 15° C. 0·900 to 0·935; and amount of phenol bodies, 20 to 30 per cent.

He has obtained the following results with different commercial oils:

No.	Kind.	Specific Gravity at 15° C.	Solubility in 80 Per Cent. Alcohol.	Phenol Bodies, Per Cent.	Optical Rotation.
1	White	0·877	Insol. in 20 vols.	2·55	—
2	,,	0·881	Insol. in 20 vols.	4·26	—
3	,,	0·863	Insol. in 10 vols.	None	—
4	,,	0·8964	Sol. in 2 vols.	4	−3°48′
5	,,	0·8935	Insol. in 10 vols.	27	−3°48′
6	Red	0·907	Sol. in 2 vols.	25·56	−1°24′
7	,,	0·880	Insol. in 10 vols.	8·73	—
8	,,	0·893	Insol. in 10 vols.	18·81	−1°6′
9	,,	0·916	Sol. in $1\frac{3}{4}$ vols.	30·16	−2°
10	,,	0·9231	Insol. in 10 vols.	19·00	—
11	,,	0·9084	Sol. in 2 vols.	14	+1°48′
12	,,	0·9074	Sol. in 2 vols.	24	−1°30′

In the author's opinion, there is but little genuine oil of thyme on the market, the white oil in particular being largely adulterated with turpentine.

Of the red oils in the above table, Nos. 6 and 12 were considered genuine.

The proportion of phenol bodies was determined by shaking 10 c.c. of the oil with a 5 per cent. solution of sodium hydroxide in a 100 c.c. nitrometer for five minutes, and then leaving it for twenty-four hours. The amount of oil freed from phenol could then be read off and the percentage calculated. C. A. M.

Looff's Process for the Estimation of Morphine in Opium. **N. A. Orlow and P. K. Horst.** (*Farmaz. J.*, 1901, xl., 91; through *Chem. Zeit. Rep.*, 1901, 105.)—This process (ANALYST, 1896, xxi., 163) gives lower results than those propounded by Dieterich and Flückiger; and the present authors have accordingly investigated all three to ascertain which gives the purest product. The figures obtained were:

	Morphine.	Nitrogen in Morphine.
By theory	—	4·91 per cent.
Looff	10·21 per cent.	4·72 ,,
Dieterich	11·71 ,,	3·57 ,,
Flückiger	11·94 ,,	3·51 ,,

F. H. L.

The Assay of Conium Seed or Leaves. H. M. Gordin. (*Amer. Journ. Pharm.*, 1901, lxxiii., 217, 218.)—Conium cannot be assayed by the author's general method (ANALYST, xxv., 74) owing to the facts that coniine is not completely precipitated by Mayer's or Wagner's reagent, and that the alkaloid is so volatile, even at the ordinary temperature, that its solutions in immiscible solvents cannot be evaporated without loss.

The following modification of Cripp's method is said to give excellent results: 20 grammes of finely-powdered conium are shaken in a 300 c.c. stoppered bottle with 200 c.c. of a previously-prepared mixture of 1 part of chloroform and 3 parts of ether for about five minutes. Ten c.c. of a 10 per cent. solution of potassium hydroxide are then introduced, and the bottle shaken at intervals for four hours and set aside over night. The next day 100 c.c. of the clear liquid are transferred, by means of a pipette, into a 300 c.c. flask, and thoroughly mixed with 10 c.c. of a 2 per cent. solution of oxalic acid in alcohol. The liquid is completely distilled off, the last traces being expelled by blowing air through the flask, which is warmed on the water-bath. After cooling, 10 c.c. of absolute alcohol are introduced, and the flask gently heated, and again cooled.

The alcoholic solution is now filtered into a wide beaker, and the flask washed out with three successive portions of 5 c.c. of absolute alcohol. The beaker is placed on a warm water-bath, and after almost complete evaporation of the alcohol, 10 c.c. of water are added, and the whole made up to 25 c.c., shaken with about 2 grammes of talc, and filtered.

12·5 c.c. of the filtrate (= 5 grammes of the drug) are then shaken in a separating funnel with 25 c.c. of petroleum spirit (boiling below 60° C.), and 5 c.c. of a 10 per cent. solution of potassium hydroxide. The lower layer is withdrawn and shaken in a second separating funnel with 20 c.c of petroleum spirit, the new lower layer drawn off into a beaker, and the upper layer transferred to the first funnel. The aqueous liquid in the beaker is again shaken with fresh quantities of petroleum spirit, until the residual liquid gives no precipitate when rendered acid and tested with Wagner's reagent.

The united petroleum spirit extracts are shaken for fifteen minutes with about 0·5 gramme of magnesium oxide, and then filtered into a 300 c.c. flask, the filter being covered with a watch-glass. The filtrate and washings are mixed with 50 c.c. of a clear saturated solution of hydrochloric acid gas in anhydrous ether, and the liquid evaporated on a water-bath, the last traces of the solvent and free acid being removed by a current of dry air.

From 25 to 30 c.c. of $\frac{N}{10}$ silver nitrate solution and 5 c.c. of 10 per cent. nitric acid are now introduced into the flask, which is warmed on the water-bath until the liquid becomes clear. The flask is then cooled, and its contents made up to 100 c.c. and filtered. The chlorine in 50 c.c. of the filtrate is determined by Volhard's method.

The number of c.c. of $\frac{N}{10}$ silver nitrate consumed by the 5 grammes of the drug, multiplied by 0·0635, gives the percentage of coniine in the sample. C. A. M.

Uganda Aloes. **A. Tschirch and J. Klaveness.** (*Arch. Pharm.*, 1901, ccxxxix., 241; through *Chem. Zeit. Rep.*, 1901, 177.)—The Uganda aloe has recently been introduced into commerce. It belongs to the Cape aloe class, and its aloin is identical with Cape-aloin. Its resin is a compound of paracumaric acid with Uganda-resinotannol, which is identical with the resinotannol of Natal aloes. The latter has the composition $C_{22}H_{22}O_8$. The Uganda aloe also contains emodin. F. H. L.

Analysis of Fluid Extract of Condurango (*Gonolbus Condurango*). **J. Warin.** (*Journ. Pharm. Chim.*, 1901, xiii., 506-512.)—This preparation is found in the Swiss and German Pharmacopœias, but not in those of England or America. It is prepared by macerating the powdered bark with a mixture of glycerin, alcohol and water, though in the author's opinion it would be advisable to replace the glycerin by alcohol and so dissolve more of the resin, which is regarded as one of the active principles.

For the analysis the author has worked out the following method: Ten c.c. of the fluid extract are mixed with 40 c.c. of water, which is sufficient to precipitate the whole of the resin. The liquid is then brought to the boiling-point to expel the alcohol. By this treatment the condurangine is also precipitated, but redissolves as the liquid cools.

The precipitate of resin is collected on a weighed filter, washed with 100 c.c. of water, dried and weighed. The filtrate is treated with 150 drops of a solution of tannin (1 : 25), and the precipitated alkaloid collected on a weighed filter, dried and weighed.

The following results were obtained with extracts prepared with alcohol of different degrees of strength, and with and without glycerin:

Preparation.	Strength of Alcohol, Per Cent.	Specific Gravity. Fresh.	Specific Gravity. After 2 Months.	Residue Dried at 100° C.	Residue, Dry or with Glycerin.	Resin, Per Cent.	Weight of Tannin Precipitate, Per Cent.
Swiss ...	22·5	1·074	1·0585	23·37	Glycerin	0·22	1·46
German ...	22·5	1·072	1·0583	23·52	,,	0·25	1·80
,, (*a*)	30	1·0298	1·0297	15·85	Dry	0·38	2·20
,, (*b*)	45	1·0122	1·0121	17·27	,,	0·56	3·65
,, (*c*)	60	—	—	17·86	,,	1·43	3·28

C. A. M.

TOXICOLOGICAL ANALYSIS.

Spectroscopic Examination of Blood. **B. Tollens.** (*Ber.*, 1901, xxxiv., 1426.)—When a blood solution is examined spectroscopically the two absorption bands of oxyhæmoglobin are quite distinct, but the single band of reduced hæmoglobin is lacking in sharpness. If the blood is treated with a little formaldehyde, the oxyhæmoglobin bands are not affected in the least, but if the liquid is then gently warmed with ammonium sulphide they gradually vanish, and almost exactly midway between

them appears a single sharp, black band. On cooling the solution and shaking it with air the single band disappears, and the pair become visible again; on warming once more the phenomena recur, and so on. In presence of carbon monoxide, formaldehyde does not produce this effect, as the two bands of carbon monoxide-hæmoglobin resist the action of the reagent. F H. L.

Recognition of Bromoform and Bromal in Cases of Poisoning. D. Vitali. (*Boll. Chim. farm.*, 1901, xl., 173; through *Chem. Zeit. Rep.*, 1901, 194.)—The material to be examined is brought with water into a flask and distilled in a current of hydrogen. The bromoform volatilizes and passes over with the gas, although its boiling-point is 151° C. If the gas is led through a tube narrowed at the extremity, ignited, and the flame allowed to play on copper gauze, the metal is coloured blue by the formation of copper bromide. If the products of combustion are drawn through weak ammonia in a Woulff's bottle and the liquid is acidified with nitric acid, the bromine may be determined with silver nitrate. If the stream of gas is led over a dry mixture of thymol and potassium hydroxide, the substance turns violet; and if an alcoholic solution of potash containing a little aniline is employed, the odour of isobenzonitrile may be detected. By causing the products of combustion to play on a basin moistened with ammonia, a white cloud of ammonium bromide is produced, which soon condenses to fine crystals.

Bromal may be recognised similarly. The material is first distilled, and the distillate is treated with potassium hydroxide, which breaks up the volatilized bromal into potassium formate and bromoform. The methods are analogous to those suggested in 1881 for the identification of chloroform and chloral. F. H. L.

ORGANIC ANALYSIS.

The Oxidation of Nitrogen as a Source of Error in the Estimation of Hydrogen and Methane. Alfred H. White. (*Journ. Amer. Chem. Soc.*, xxiii., 477.)—The author found that oxides of nitrogen are always formed in explosion analysis, the amount increasing with the violence of the explosion, and that the dilution recommended by Bunsen when working with cylindrical eudiometer tubes under reduced pressure is not sufficient when working with the spherical Hempel pipettes, in which, since the explosion is more rapid, and the walls take up less of the heat generated, the temperature reached is higher. If the explosive ratio (ratio of inert to explosive gas) is kept between three and four, the error is negligible in the technical analysis of gases consisting almost entirely of hydrogen, but increases with the quantity of methane present, and may amount to more than 1 per cent. The method of Dennis and Hopkins (*Journ. Amer. Chem. Soc.*, xxi., 398), in which the gas is burnt quietly in a current of air and oxygen by means of a glowing platinum spiral tends to give high results, and care must be taken that the wire is not heated too much. Apparently no method involving active combustion can give strictly accurate results. A. G. L.

Spectroscopic Detection of Methylfurfurol. K. Oshima and B. Tollens. (*Ber.*, 1901, xxxiv., 1425.)—Widtsoe and Tollens have already shown that methylfurfurol derived from the methylpentosans and methylpentoses of natural products gives a characteristic absorption band in the spectroscope. The present authors have discovered that an addition of phloroglucinol renders the test more delicate and noticeable. The organic matter is distilled with 1·06 hydrochloric acid. Five c.c. of the distillate are treated with an equal volume of strong hydrochloric acid, and a little of a solution of phloroglucinol in 1·06 HCl is added. A red precipitate of methylfurfurolphloroglucide falls, which is filtered off after five minutes' standing; the filtrate is coloured orange, and before the spectroscope shows a dark band at the beginning of the blue, while the violet is partially obscured. F. H. L.

The Detection and Estimation of Artificial (Cellulose) Silk in Fabrics. M. Duyk. (*Bull. de l'Assoc. Belge*, 1901, xv., 166-175.)—The fibres are first treated in the cold with a 5 per cent. solution of sodium hydroxide, to which are added a few drops of a solution of sodium hypochlorite, if required. They are then immersed in dilute hydrochloric acid (1 : 10), washed with water, pressed between filter-paper, and dried at a gentle heat. They are subjected to the following tests:

Combustion.—Artificial silk burns readily without emitting the odour given by animal fibres, and leaves a white ash.

Action of Sodium Hydroxide (*2 per cent. solution*).—Artificial silk is scarcely attacked, whereas natural animal fibres are dissolved more or less readily.

Action of Concentrated Sodium Hydroxide.—When immersed in a solution of specific gravity 1·33, fibres derived from cellulose are converted into a gelatinous mass.

Action of Nitric Acid.—Natural animal fibres are coloured yellow through the formation of xanthoproteic acid, whilst artificial silk is decomposed and frequently falls to powder.

Millon's Reagent.—On immersing the decolorized fibres in the boiling reagent, artificial silk does not alter, whereas natural silk acquires a deep red colour and wool a reddish-yellow colour.

Pyroxyline Test.—The following test is prescribed by the French Customs authorities: A little of the material is freed from any dressing, dried, and immersed in a mixture of 2 parts of sulphuric acid (specific gravity 1·835) and 1 part of nitric acid (specific gravity 1·50) at a temperature of about 25° C., and left for an hour. Natural silk is dissolved, whilst artificial silk is nitrated. In the latter event the fibres are thoroughly washed, dried, and identified by their behaviour on ignition and solubility in acetone.

In the author's experience this method is not altogether satisfactory, for it does not distinguish between artificial silk and other fibres of vegetable origin.

Ammoniacal Solution of Nickel Carbonate.—This dissolves the fibres of natural silk, but is practically without action on other animal or vegetable fibres. The reagent is prepared by dissolving 1 part of nickel carbonate in 6 parts of ammonium hydroxide, and diluting the solution with 6 parts of water. The fibre is immersed for a few minutes in the cold liquid. After some time wool, too, is slightly attacked.

Concentrated Sulphuric Acid.—Artificial silk is dissolved. Chardonnet's artificial silk when thus dissolved gives the reaction for nitrates with diphenylamine sulphate.

Microscopical Examination.—Apart from characteristic differences in the appearance of the different fibres, which are illustrated by figures, the author recommends the following micro-chemical tests :

Iodine Water and Dilute Sulphuric Acid.—Natural silk is coloured yellow, whilst artificial silk assumes a deep blue colour. Other vegetable fibres take various colorations, from bright blue (cotton, flax) to yellowish-red (jute, etc.).

Ammoniacal Solution of Copper. — This dissolves cellulose-silk almost instantly, apparently without first causing it to swell up, as in the case of natural vegetable fibres, which are covered with a ligneous cuticle.

Aniline Hydrochloride.—A 2 per cent. solution of this salt gives a yellow or brown coloration with natural vegetable fibres, but not with artificial cellulose-silk. The fibre of *asclepias* (vegetable silk) acquires an intense yellow colour under these conditions.

Quantitative Analysis.—A weighed quantity of the material is freed from dressing and immersed in the author's ammoniacal nickel reagent. The blue solution is rapidly decolorized, and if necessary more must be added. After thirty minutes the fibres are washed with water, then with water slightly acidulated, and again with water, and dried at 105° C., and weighed.

When wool is present the older method of boiling with a 2 per cent. solution of sodium hydroxide is employed. C. A. M.

A New Method of Estimating Phenols. E. Riegler. (*Bull. Soc. Bucuresci*, viii., 51 ; through *Ann. de Chim. anal.*, 1901, vi., 231.)—This is based on the property possessed by phenols of forming a colouring matter with para-diazo-nitraniline, as in the equation

$$C_6H_4\begin{matrix}N=NCl\\NO_2\end{matrix} + C_6H_5OH + 2NaOH = NaCl + 2H_2O + C_6H_4\begin{matrix}N=N-C_6H_5ONa\\NO_2\end{matrix}$$

On adding sulphuric acid drop by drop to the alkaline solution, the colouring matter separates out in the form of the compound

$$C_6H_4\begin{matrix}N=N-C_6H_4OH\\NO_2\end{matrix}$$

This substance, which is nearly insoluble in water, is collected on a filter and weighed. The weight multiplied by the factor 0·3868 gives the corresponding amount of phenol. As a correction for the slight solubility 0·002 gramme is added to the weight found, for each 100 c.c. of liquid used. The author has used the same method with satisfactory results in the determination of thymol and guaiacol. C. A. M.

On the Elimination and Quantitative Estimation of Water in Oils, Fats, and Waxes. Charles B. Davis. (*Journ. Amer. Chem. Soc.*, xxiii., 487.)—The drying may be very readily and safely effected as follows : Into a wide-mouthed weighing bottle sufficient thick filter-paper (in coil form) is introduced to half fill the

vessel. Bottle and paper are then dried at 110° C. to constant weight, and as much of the sample is added as will just saturate the filter-paper. The increase in weight of the bottle gives the quantity taken. The whole is then dried at 110° C., if necessary in an atmosphere of carbon dioxide or hydrogen, and again weighed, the loss giving the water evaporated.

In some cases it may be more convenient to introduce the oil in ethereal solution, in which case the ether is first evaporated off at a slightly elevated temperature, and the sample then dried as above. Solid fats and waxes are added in their natural state, and are quickly absorbed by the paper after melting. The substance is easily recovered by extracting the paper in a Soxhlet apparatus. A. G. L.

A Siccative for Oil-colours in Tubes. Moriz Kitt. (*Chem. Revue über d. Fett-u-Harz-Ind.*, viii., 157.)—It has been customary for a long time to add 5 to 10 per cent. of wax to many oil-colours as a thickening agent, in order to prevent the heavier pigments from separating out. Several objections are urged by painters against the use of wax for this purpose, and a material more suitable appears to consist in cold-drawn wood oil, which has turned solid through exposure to light for a few weeks. A number of experiments were made which show that wood oil has the further advantage of causing the colour to dry more quickly. A. G. L.

The Separation of Cholesterin and Phytosterin from Mixtures of Fatty and Mineral Oils. J. Marcusson. (*Chem. Revue über d. Fett-u-Harz-Ind.*, 1901, viii., 104-105.)—In the case of thin mineral oils which are volatile with superheated steam the following method has given satisfactory results in the author's hands: From 100 to 200 grammes of the oil are saponified with alcoholic alkali, and the unsaponifiable matter extracted with ether from the soap solution, and distilled in a current of superheated steam until only about 3 c.c. of residue was left. In the distillation care must be taken that the temperature of the oil never exceeds 200° C.

The residue from the distillation is boiled with 70 per cent. alcohol, and the crystals of cholesterin or phytosterin obtained from the extract are purified by recrystallization in the usual way.

When the mixture contains a heavy mineral oil, the preceding method is not applicable, and in such cases the following method is recommended: The oil under examination is dissolved in ether, and the greater proportion of the mineral oil precipitated by the addition of an equal volume of alcohol. The clear supernatant liquid is poured off, concentrated to about 50 c.c., and, after cooling, again decanted from any further deposit, and evaporated. The residue is saponified, and the aqueous soap solution extracted several times with ether. The residue from this extract is then crystallized from 70 per cent. alcohol, as in the first method.

C. A. M.

INORGANIC ANALYSIS.

Examination of Commercial Copper Oxide. D. Miklosich. (*Zeits. f. angew. Chem.*, 1901, 753.)—Commercial cupric oxide is liable to contain cuprous oxide, which greatly diminishes its value for colouring glass green. For the estimation of this impurity advantage may be taken of the reaction :

$$Cu_2O + Fe_2O_3 = 2CuO + 2FeO.$$

One gramme of ferrous ammonium sulphate is weighed out, dissolved in dilute sulphuric acid, and titrated with permanganate. Thus, the iron is all converted to the ferric state, and the strength of the permanganate solution is simultaneously determined. Ten c.c. of dilute sulphuric acid are then added, and also $\frac{1}{2}$ gramme of the copper oxide, which is brought into solution by gently warming, this operation being carried out in a flask provided with a Bunsen valve to prevent oxidation. When cold the contents are transferred to a porcelain basin containing a litre of air-free water, and rapidly titrated with the permanganate solution.

Volumetric Estimation of Lead Peroxide in Red-Lead. M. Liebig. (*Zeits. f. angew. Chem.*, 1901, 828.)—The red-lead is reduced to a fine state of division, and 0·5 gramme is washed into an Erlenmeyer flask with a little distilled water. To this 25 c.c. of $\frac{N}{10}$ sodium thiosulphate are added, then 10 c.c. of approximately 30 per cent. acetic acid, and dissolution is brought about by shaking. Then 10 c.c. of potassium iodide solution (1 : 10) are added, and 2 to 3 c.c. of zinc iodide and starch solution, and the excess of thiosulphate is titrated with $\frac{N}{10}$ iodine solution. The number of c.c. of the thiosulphate solution oxidized by the red-lead, multiplied by 239, the molecular weight of the peroxide, gives the percentage of the latter. The end of the reaction is shown by the precipitated yellow lead iodide appearing dirty yellow. A. M.

Iodometric Estimation of Antimonic Acid. Volumetric Determination of Antimony in Presence of Tin. M. Rohmer. (*Ber.*, 1901, xxxiv., 1565.)—Although antimonic acid is not quantitatively reduced by sulphurous acid at the boiling-point, the reaction succeeds in the presence of hydrobromic acid. Based on this fact, the following process has been elaborated: The original solution, containing antimonic and hydrochloric acids, is boiled in a round-bottomed flask with 1 gramme of potassium bromide, some aqueous sulphurous acid, and a few pieces of pumice, till the odour of SO_2 has disappeared. The liquid is cooled, treated with tartaric acid, neutralized with sodium bicarbonate, and titrated with $\frac{N}{20}$ iodine. This is best effected by adding about 1 c.c. more than the necessary quantity, and titrating back with thiosulphate of equivalent strength.

Tin hinders the oxidation of antimonous to antimonic acid, but that effect is overcome by the addition of large amounts of tartaric acid. Thus, a mixture of antimony and tin can be satisfactorily separated in the following manner: The mixed sulphides which have been obtained in the course of an ordinary analysis are brought into a round-bottomed flask, and boiled with strong hydrochloric acid and

potassium chlorate till the odour of chlorine is no longer noticeable. One gramme of potassium bromide and some sulphurous acid are introduced, and the excess of the latter is boiled off. After cooling, a considerable quantity (10 to 20 grammes) of tartaric acid is added, and the liquid titrated as above. Alloys may be dissolved directly in aqua regia or in hydrochloric acid and chlorate. Antimony-tin-arsenic alloys are first treated with the latter solvent as usual, then bromide is added, and the arsenic is driven off by distillation in a current of sulphur dioxide and hydrochloric acid. F. H. L.

Modified Williams Method for Manganese. Randolph Bolling. (*Journ. Amer. Chem. Soc.*, xxiii., 493.)—In the following modification of the Williams' method, the time necessary for the filtration of the manganese dioxide is greatly shortened by the preliminary removal of the silica: 5 grammes of pig-iron drillings are treated in a beaker with 75 c.c. nitric acid (specific gravity 1·42), and, after violent action has ceased, 10 c.c. hydrofluoric acid are added. The solution is evaporated till ferric oxide begins to separate out, cooled, and heated to boiling after the addition of 100 c.c. nitric acid (specific gravity 1·42). Some asbestos fibre and then about 7 grammes potassium chlorate are added, and the solution is boiled until the green fumes have disappeared, cooled rapidly, and filtered through asbestos. After two washings with nitric acid, the precipitate is washed with cold water until the washings are neutral to litmus. The asbestos pad and precipitate are then transferred to the original beaker, 50 c.c. of standard acid ferrous sulphate solution run in, and after the precipitate is entirely dissolved the excess of ferrous sulphate is titrated with decinormal permanganate solution.

The solutions should be standardized by treating a sample of pig-iron with a known percentage of manganese as above, and, after the titration is finished, running in a second quantity of 50 c.c. of the iron solution, and titrating this with the permanganate. By this method of standardization the errors due to the asbestos and carbon floating about in the liquid, as well as those due to irregularity in the precipitate, are avoided. Results obtained by this method agreed very well with those obtained by the Gibbs' method, in which the manganese is weighed as pyrophosphate, the largest deviation being 0·02 per cent. A. G. L.

The Volumetric Determination of Zinc. Percy H. Walker. (*Journ. Amer. Chem. Soc.*, xxiii., 468.) — The following method is an application of Handy's modification of Stolba's method for the estimation of magnesium (*Journ. Amer. Chem. Soc.*, xxii., 31) to the case of zinc: To the zinc solution, which must contain ammonium chloride, a large excess of ammonia is added, and then a large excess of sodium phosphate. No precipitate is formed, but on cautiously neutralizing the excess of ammonia with dilute acid, the liquid gradually becomes milky, when it should be heated to 75° C., and the neutralization continued, with continuous stirring, until a piece of litmus-paper placed in the solution just turns violet, whilst there is no smell of ammonia. The crystalline precipitate is filtered after five minutes' standing and washed with cold water; paper and precipitate are returned

to the beaker in which precipitation was effected, an excess of standard acid and a few drops of methyl orange added, and the excess is determined by standard alkali. According to the equation,

$$Zn(NH_4)PO_4 + H_2SO_4 = ZnSO_4 + (NH_4)H_2PO_4,$$

1 c.c. of normal acid corresponds to 32·7 milligrammes of zinc.

In three determinations by this method the average error was −0·0006 gramme on 0·1490 gramme zinc taken. The method may be used in the presence of magnesium, ferric iron, and calcium, since the phosphates of these three metals are precipitated in a strongly ammoniacal solution, and can be removed by filtration before neutralizing the liquid. In two determinations, in which all three metals were present, the error was −0·0020 gramme on 0·1192 gramme zinc taken. Manganese, if present, must be previously separated. A. G. L.

Determination of Zinc in Spathic Iron Ore. J. Flath. (*Chem. Zeit.*, 1901, xxv., 564.)—This process is specially suitable for the determination of small quantities of zinc (0·4 to 5·0 per cent.) in presence of much iron and manganese. Three to 5 grammes of material are dissolved by gentle warming in hydrochloric acid, diluted with 150 or 200 c.c. of water, and treated with a small excess of ammonia. Without filtering, the precipitate is dissolved in 15 c.c. of 96 per cent. acetic acid, and sulphuretted hydrogen is passed through the liquid to throw down all zinc, lead, copper, and 2 or 4 per cent. of the iron. This precipitate settles very quickly, which zinc sulphide alone does not; and, by avoiding access of air as far as practicable, it can be filtered in five or ten minutes' time. The filtrate is perfectly free from zinc: it always becomes milky, but from presence of sulphur only. The mixed sulphides are washed twice with sulphuretted hydrogen water, dissolved in aqua regia, evaporated with 10 c.c. of 1 : 1 sulphuric acid, diluted with 100 c.c. of water, boiled, and the liquid treated with 10 c.c. of 10 per cent. sodium thiosulphate to remove the copper. Five c.c. of nitric acid are added to the filtrate in order to destroy the excess of thiosulphate and to oxidise the iron, and the whole is somewhat concentrated. The manganese is next thrown down by two precipitations with bromine and ammonia, and in the filtrate the zinc is estimated volumetrically.

The foregoing method has the advantage of permitting 3 or 4 grammes of substance to be analysed without danger of losing zinc in the usual iron precipitate. The tedious and complicated precipitation of iron and manganese together is also wholly avoided. It yields higher results in zinc than the ordinary processes; and experiments on mixtures of spathic iron ore (free from zinc) with known quantities of a pure zinc salt show it to be more accurate. F. H. L.

On the Separation of Tungstic and Silicic Acids. H. L. Wells and F. J. Metzger. (*Journ. Amer. Chem. Soc.*, xxiii., 356.)—Otto Herting has recently stated (*Zeits. angew. Chem.*, 1901, 165) that the well-known method of expelling silica from tungstic acid by means of hydrofluoric acid is incorrect, since a part of the tungstic acid also volatilizes. The authors have examined the behaviour of mixtures of pure

silica and tungstic acid when treated, before and after ignition over a Bunsen burner, with a mixture of hydrofluoric and sulphuric acids, and with hydrofluoric acid alone, followed by evaporation to dryness and ignition of the residue over a Bunsen burner. In all cases the silica was expelled quantitatively, the greatest alteration in the weight of the tungstic acid taken being 0·0001 gramme. The method thus appears to be perfectly exact, and the authors ascribe Herting's results to the fact that tungstic acid itself volatilizes slowly when ignited over the blast-lamp, and that he probably ignited his tungstic acid at too high a temperature. A. G. L.

The Detection of Selenium in Sulphuric Acid. A. Jouve. (*Bull. Soc. Chim.*, 1901, xxv., 489-491.)—After referring to the codeine and sulphurous acid tests for selenious acid, the author points out that selenic acid is only slowly acted upon by sulphur dioxide, and does not give the codeine reaction. He finds that crude acetylene gives more or less pronounced red coloration when passed into sulphuric acid containing selenium, and that it will detect 1 part in 100,000.

The reaction is partially due to the reduction of the selenium compounds by impurities in the crude acetylene, although the pure gas is the principal agent. The speed of the coloration is increased by the presence of hydrochloric acid vapours.

C. A. M.

The Estimation of Carbonic Acid in Water. Joseph W. Ellms and Jay C. Beneker. (*Journ. Amer. Chem. Soc.*, xxiii., 405.)—As the result of an investigation into the comparative merits of the Pettenkofer, Trillich's modified Pettenkofer, and the Lunge-Trillich or Seyler methods (ANALYST, xxii., 312), the authors conclude that the last is the most accurate and rapid, the error on the "free and half-bound carbonic acid" being on an average less than 1 per cent., with a possible range of less than ±3 per cent. In titrating the "fixed" carbonic acid according to Hehner's method, the authors prefer the use of lacmoid to methyl orange, which is used by Seyler. Trillich's modification of Pettenkofer's method gives results which are generally 5 per cent., and may be as much as 12 per cent., too low. Pettenkofer's method gives still lower and less uniform values, and under some conditions it yields extremely erratic results. A. G. L.

The Detection and Estimation of Nitrates in Water. P. Cazeneuve and H. Défournel. (*Bull. Soc. Chim.*, 1901, xxv., 639, 640.)—The authors' method is based upon the fact that a blood-red coloration is produced when a dry mixture of a nitrate and brucine is treated with glacial formic acid.

The residue obtained from the evaporation of a litre of the water is taken up with 20 c.c. of water and the solution evaporated in a small flat-bottomed dish on the water-bath with 0·05 gramme of brucine. A few drops of glacial formic acid are then added to the warm residue, and immediately afterwards a little water. A yellow tint, changing after twelve hours to rose, is obtained in the presence of a nitrate. The addition of hydrogen peroxide effects the change to rose in fifteen minutes. The limit of sensitiveness of the reaction is 1 : 100,000.

Unlike the colour produced by sulphuric acid and brucine with nitrates, which is fugitive, the rose coloration in this test remains unchanged for several days.

The test is made quantitative by comparing the colour with that obtained with a dry residue containing a known quantity of nitrate. C. A. M.

REVIEW.

HANDBOOK ON PETROLEUM. By Captain J. H. THOMSON and BOVERTON REDWOOD. London: Chas. Griffin and Co., Ltd. Price 8s. 6d. net.

The authors have done excellent service in writing this handbook, which contains in moderate compass and in very clear language a mass of practical information concerning petroleum, calcium carbide, and acetylene, and the precautions necessary for their safe conveyance, storage, and employment, which will be valuable to all who have to deal with these products, whether as inspectors under the Petroleum Acts, traders, or users. At the outset the authors explain that the legal definition of "petroleum" is a wide one, embracing many liquids and manufactured products which are not petroleum in the commercial sense; at the same time, the bulk of manufactured petroleum is not included in it, but only such as flashes below 73° F. They then give a brief history of petroleum, and a useful epitome of the theories concerning it, the most probable being that it is derived from organic remains, partly animal and partly vegetable.

Chapters II. and III. contain an account of the sources of supply and their relative importance; a detailed description of the petroleum well and its construction (which suffers in clearness owing to the absence of illustrations); the means employed for transport, storage, and distribution; and an outline of the refining processes. To supply the world's present consumption of petroleum, a pipe 41 inches in diameter would be required, assuming the rate of flow to be 3 feet per second.

In Chapter IV. the various commercial products obtained from petroleum, shale oil, and coal-tar are described, with their names and uses, specific gravities, flash-points, and boiling-points. This chapter will be useful to many, as there is a good deal of confusion respecting the precise significance of some of these commercial names. In the next chapter the meaning of "flash-point" and "fire-test" are explained in the clearest possible manner, and the conditions which influence the flash-point are discussed in detail.

With regard to the much-debated question whether the present legal flash-point of 73° F. needs alteration, the authors deprecate the basing of conclusions upon such considerations as the maximum atmospheric temperature, or the maximum temperature of the oil in any lamp, because the conditions of the test are not the same as those which arise in actual practice. They consider that the standard of flash-point should be judged by experience alone, and that no alteration should be made on merely theoretical grounds. "The real question is whether oil of a given flash-point has shown itself to be so dangerous by reason of its volatility as to require the standard to be raised, and, if so, whether the advantage in point of safety outweighs

any disadvantage which may be involved." Statistics are quoted to show that the number of fatal lamp-accidents is not so great as to call for legislation of a very drastic character; in fact, more than five times as many fatalities are caused by people falling downstairs. If the flash-point were to be raised above the highest temperature which may occur in an ordinary lamp, it would have to be raised to at least 150° F. The British public, however, could not be compelled to use oil of this high test, and if oil of lower flash-point were found to be as good or better, and cheaper, it would continue to be used. The authors evidently consider any alteration of the existing flash-point both unnecessary and undesirable. They consider that 90 per cent. of the accidents which have occurred have been due to ignorance, carelessness, slovenliness, or false economy in purchasing flimsy lamps, and they look for the remedy to a better education of the people.

In Chapter VI. methods for determining the flash-point are detailed, with a history of the evolution of the Abel test. The Abel-Pensky test is very fully illustrated and described, "as advantage will probably be taken of the first opportunity to legalize its use in this country." The German instructions are given in full; also a table for correction of observed flashing-points for variations in barometric pressure, which, though not yet legalized, it is suggested that inspectors should make use of.

The bungling history of petroleum legislation is recounted in Chapter VII. In a fortunately abortive Bill introduced in 1869 nitroglycerin was included, "to which, apparently, the officer of the local authority was called upon to apply the flash-test"! The existing legislation is summarized in Chapter VIII., and the directions in which amendment and further legislation are desirable are pointed out.

In Chapters IX. and X. the precautions necessary to avoid accident in the conveyance, storage, and use of petroleum are fully discussed, and valuable suggestions are given as to the construction and use of mineral oil lamps, based upon the evidence given before the Select Committee on Petroleum. The last chapter is devoted to calcium carbide and acetylene. Sixteen appendices, containing the Petroleum Acts, Orders in Council, Model Rules for the Care and Use of Petroleum Lamps, etc., and an index complete the work.

It is to be hoped that the authors' wish may be realized, and that the general voluntary adoption of the safeguards which they have shown to be necessary may anticipate and even render unnecessary further legislation. To this end their handbook cannot be too widely read, and it should be possessed by all who have to advise professionally on matters relating to petroleum. L. A.

INSTITUTE OF CHEMISTRY.

We are informed that the next Examination in Therapeutics, Pharmacology, and Microscopy will be held at 30, Bloomsbury Square, London, W.C., in January, 1902; also that the exact date is not yet fixed, but will be communicated to intending candidates. Candidates desirous of presenting themselves are required to send in their applications on or before Tuesday, the 3rd day of December, 1901.